

9th Eastern European Young Water Professionals Conference



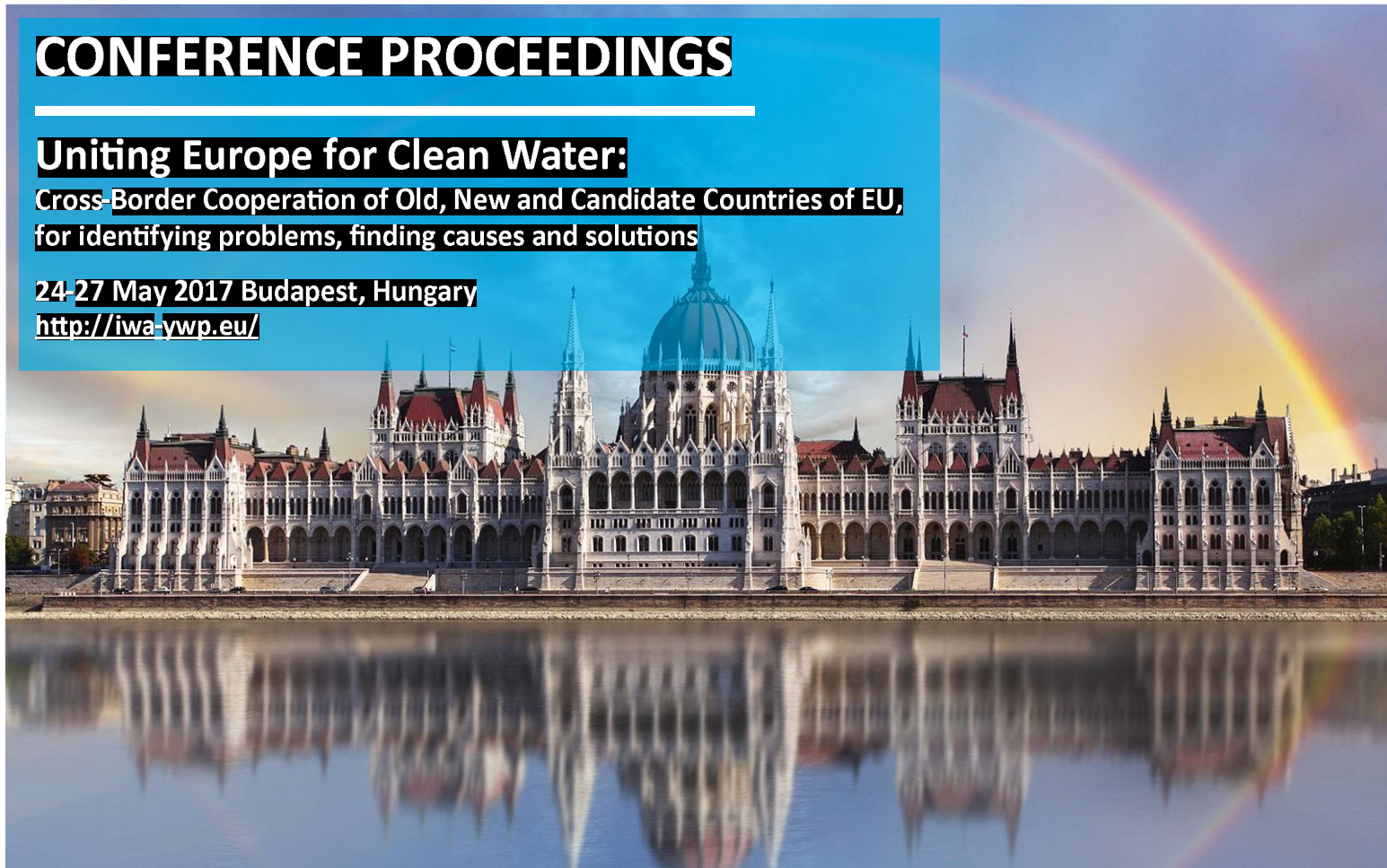
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Uniting Europe for Clean Water:

Cross-Border Cooperation of Old, New and Candidate Countries of EU,
for identifying problems, finding causes and solutions

24-27 May 2017 Budapest, Hungary

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Editors:

Maryna Feierabend

Olha Novytska

Vince Bakos

Typesetting:

Maria Danilycheva

Maja Djogo

Éva Zofia Sebestyén

Cover Design:

Maryna Feierabend

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Dielectric Constant Measurement in Wastewater Treatment Processes

S. Beszédes, Sz. Kertész, B. Lemmer, I. Kovács, G. Keszthelyi-Szabó and C. Hodúr*

* Department of Process Engineering, Faculty of Engineering, University of Szeged, H-6725 Szeged, Moszkvai krt. 9., Hungary (E-mail: beszedes@mk.u-szeged.hu)

Abstract

Numerous studies have been published in the last years investigating the applicability of the microwave pre-treatments in wastewater and mainly in sludge utilization technologies. It has been verified that microwave irradiation, as pre-treatment method, could be advantageous if organic matter fraction is utilized in further biological processes. Because of the change of compounds and the physicochemical structure of organic matter fraction, the dielectric parameters can be changed during the pre-treatment process. Because there are very few data available related to dielectric parameters of different originated wastewater, the objective of our work was to measure the dielectric constant during the purification process of municipal wastewater and pre-treatment of meat- and dairy industry wastewater in static and continuously flow operation mode.

Keywords

Wastewater; sludge; dielectric properties; microwave

INTRODUCTION

Degree of hydrolysis is one of the key issues for the whole efficiency of the sludge utilization processes. In hydrolysis the polymeric and insoluble compounds, such as proteins, fats, carbohydrates and their derivatives are decomposed into less molecules, such as amino acids, monosaccharides, alcohols or fatty acids. Due to the complex particle structure, and presence of strong cell membranes sludge is difficult to directly bio-degrade under aerobic or anaerobic conditions, as well. The main aim of sludge pre-treatment technologies is to disrupt the cell membranes, thus lysis of the cells of microorganisms, and to accelerate the hydrolysis of macromolecular components. Methods for sludge disintegration can be based on mechanical, thermal and chemical processes or their combination.

Among the different sludge handling methods, microwave irradiation is successfully applied in the process of hygienization, dewatering, drying and pre-treatment stage of anaerobic digestion (AD). Pre-treatments by microwave irradiation offers advantages such as rapid and direct heating, reduction of energy loss, better controllability compared to the conventional thermal methods. Higher energetic efficiency of dielectric heating based in the different way of energy transfer than conventional heating. In conventional heating the electric energy is converted into heat, after the heat is transferred to the surface of the material, and the heat penetrates from the surface towards the core of materials. During dielectric heating microwave energy is delivered into the material directly, and heating occurs inside of the materials (volumetric heating) due to the molecular interactions with electromagnetic field (Appleton et al., 2005).

A microwave method is suitable to increase the degree of conversion of organic matters in the sludge flocks into easily accessible compounds for fermentative microorganisms. Disruption of cell membranes led to release of the protoplasm-enzymes responsible for the increasing of ammonia and phosphate concentration in sludge liquor. Considering the efficiency of sludge disintegration and

the biogas production kinetic, microwave pre-treatments has been found to be superior to conventional thermal pre-treatment processes.

The most used method to characterize the change in the efficiency of disintegration is the measure of the soluble to total chemical oxygen demand (SCOD/TCOD) (Tyagi and Lo, 2013). Lysis of microbial cell walls and disintegration of sludge flocks is determined by hydrogen bindings, hydrophobic interactions and also by the concentration of divalent cations. Increment of Ca^{2+} and Mg^{2+} , as a component of phospholipids, is due to the disruption of cell walls (Ahn et al., 2009). Therefore the measurement of divalent ion concentration in liquid phase can be suitable indirect method to follow the degree of disintegration. Furthermore, the strong effect of microwave irradiation on the cell wall disruption and sludge flock disintegration was confirmed by microscope observations (Bohdziewicz et al., 2006), as well.

Results of preliminary studies established that organic matter releasing is mainly influenced by the final temperature and temperature ramp of sample during the process and the duration of microwave irradiation. If the final temperature is higher than 80°C a partial conversion of NH_4^+ into gas phase as NH_3 can be observed, decreasing the organic matter concentration of liquid phase (Eskicioglu et al., 2007). Furthermore, if the temperature of sample reach the boiling point a slight decrease of COD concentration occurred (Bohdziewicz et al., 2011). Because the heat stress is influenced by the temperature ramp which is depended on the strength of electromagnetic field, the power of microwave irradiation can be also determinative process parameter (Beszedes et al., 2011).

Numerous reports concluded that microwave pre-treatment is suitable to enhance the efficiency of anaerobic digestion process, what manifested in higher biogas product and accelerated biogas production rate (Bougrier, 2007). The increment of biogas production and utilization value of biogas (calorific value and quality determined mainly by the methane, carbon dioxide and hydrogen sulfide concentration) is affected by the type of processed sludge.

Thermal and athermal effects of microwave irradiation have been investigated for many years. Athermal effects means a special effect of electromagnetic field at microwave frequency range that is not associated with temperature increasing of irradiated materials. Microwave radiation can polarize the side chains of macromolecules, and a change in dipole orientation of polar molecules can be occurred. High frequency electromagnetic waves induce structural changes and orienting effects within the materials. It can be noticed, that at frequencies of 915 MHz and 2450 MHz, which are commonly used for industrial and scientific applications, the energy of microwave photon is not enough to break hydrogen and covalent bonds, or induce directly chemical reactions (Kappe, 2004). Heat generation efficiency of microwaves is determined by the frequency, concentration, structure and composition of irradiated matrix, particle size, viscosity, individual and integral dielectric properties of compounds, penetration depth etc. Heating mechanisms of microwave irradiation can be attributed by dielectric polarization and ionic conduction. Depend to the applied frequency, dipolar molecules, such as water; move in time with the varying polarity electromagnetic field. During microwave irradiation local charges of dipolar materials tend to move. If dipole lag behind the electromagnetic field energy dissipation is occurred in the material led to heat generation. In wastewater and sludge water (moisture) can be considered as the main component. Dipole movement was influenced by the strength of hydrogen bounded matrix of materials. In the case of the free water content the movement has been revealed at GHz frequency range. Dipolar movement is the dominant mechanism for bound water at MHz frequency ranges, for ice at kHz frequencies (Brodie et al., 2014).

In heterogeneous and complex systems the dielectric properties are determined by the molecular structure of compounds, and the physico-chemical properties, as well. The continuous realignment of polarized dipoles to the varying polarity electromagnetic field can result in collisions of dipolar molecules and weakening of hydrogen bonds within water molecules (Afolabi and Sohail, 2017). Behavior of molecules in the irradiated materials is characterized by the dielectric parameters, such as the complex dielectric permittivity or its real and imaginary component i.e. the dielectric constant and dielectric loss factor. Dielectric loss factor indicates the transmission loss (power dissipation) in material, i.e. the conversion of irradiated microwave energy to heat. Dielectric constant characterized the capacity properties of materials, the ability of the dipolar molecules for polarization at a given frequency.

Because the dielectric parameters are linked to the molecular structure of materials, change in physiochemical structure can be detected by dielectric measurements. On the other hand, because the change of dielectric parameters has effect on heating efficiency of microwaves, structural change in processed materials has a retroaction for the efficacy and economy of microwave heating processes. It has been verified elsewhere, that microwave pre-treatments are suitable for preliminary hydrolysis of macromolecules before anaerobic digestion of sludge, what led to increasing in biogas production. During microwave pre-treatment carbohydrates are hydrolyzed into sugars; proteins into amino acids, or lipids into short-chain fatty acids. With decreasing of molecular weight and increasing of polar characteristic of hydrolyzed monomers, the polarization ability of the processed materials enhanced, therefore these changes can be characterized and quantified by the change of dielectric parameters.

MATERIALS AND METHODS

Municipal wastewater samples were originated from a local wastewater treatment plant, purified wastewater was sampled after biological stage of WWTP. Raw (untreated) food industry wastewater sampled from the puffer tank of wastewater line of a meat processing plant. Main characteristics of samples are summarized in Table 1.

Table 1. Main characteristics of wastewater samples

Parameter	Unit	Municipal wastewater		Meat industry wastewater
		raw	purified	
pH	[-]	7.3 ± 0.3	7.1 ± 0.2	5.9 ± 0.2
TCOD	[mgO ₂ L ⁻¹]	986 ± 34	102 ± 2.1	1697 ± 93
SCOD	[mgO ₂ L ⁻¹]	115 ± 22	68 ± 4.1	323 ± 11.8

Total solid content (TS) of samples was determined by drying cabinet method using temperature of 105°C to achieve constant weight. Organic matter content was measured by COD fractionation method. Soluble chemical oxygen demand (SCOD) was measured using colorimetric method (APHA, 2005) from supernatant of samples after centrifugation (10000 rpm for 15 minutes) and microfiltration (0.45 µm). Total chemical oxygen demand (TCOD) was measured from whole matrix after dilution. Disintegration degree (DD%) was given as the change in the ratio of SCOD to TCOD.

Microwave pre-treatments was carried out in a tailor made microwave unit equipped with variable power magnetron (from 100 W to 700W) operating at 2450 MHz frequency. In continuously flow operation mode, the volumetric flow rate of irradiated samples was varied by the rpm of peristaltic pump in the range of 10-45 Lh⁻¹.

Dielectric constant (ϵ') was determined in a tailor made dielectrometer equipped with a dual channel NRVD power meter (Rohde & Schwarz). Magnetron of dielectrometer operates at a frequency of 2450 MHz. ϵ' was calculated from the reflection coefficient (Γ) phase shift (ϕ) according to the (1) and (2) equations

$$\delta = \arctg\left(\frac{|\Gamma|\sin\phi}{1-|\Gamma|\cos\phi}\right) - \arctg\left(\frac{|\Gamma|\sin\phi}{1+|\Gamma|\cos\phi}\right) \quad (1)$$

$$\epsilon' = \frac{1}{\sqrt{1+\tan^2\delta}} \left(\frac{1+|\Gamma|^2+2|\Gamma|\cos\phi}{1+|\Gamma|^2-2|\Gamma|\cos\phi} \right) \quad (2)$$

Central composite face centered (CCF) experimental design and response surface methodology (RSM) was applied to investigate the effect of variables on target parameters using STATISTICA 13 software.

RESULTS AND DISCUSSION

In the first series of experiments the effect of total solids (TS) and temperature on disintegration degree (DD%) (Figure 1a) and dielectric constant (Figure 1b) were investigated for municipal wastewater sludge. TS content was adjusted by the mixing of wastewater and sludge originated from municipal wastewater produced in sedimentation process at WWTP.

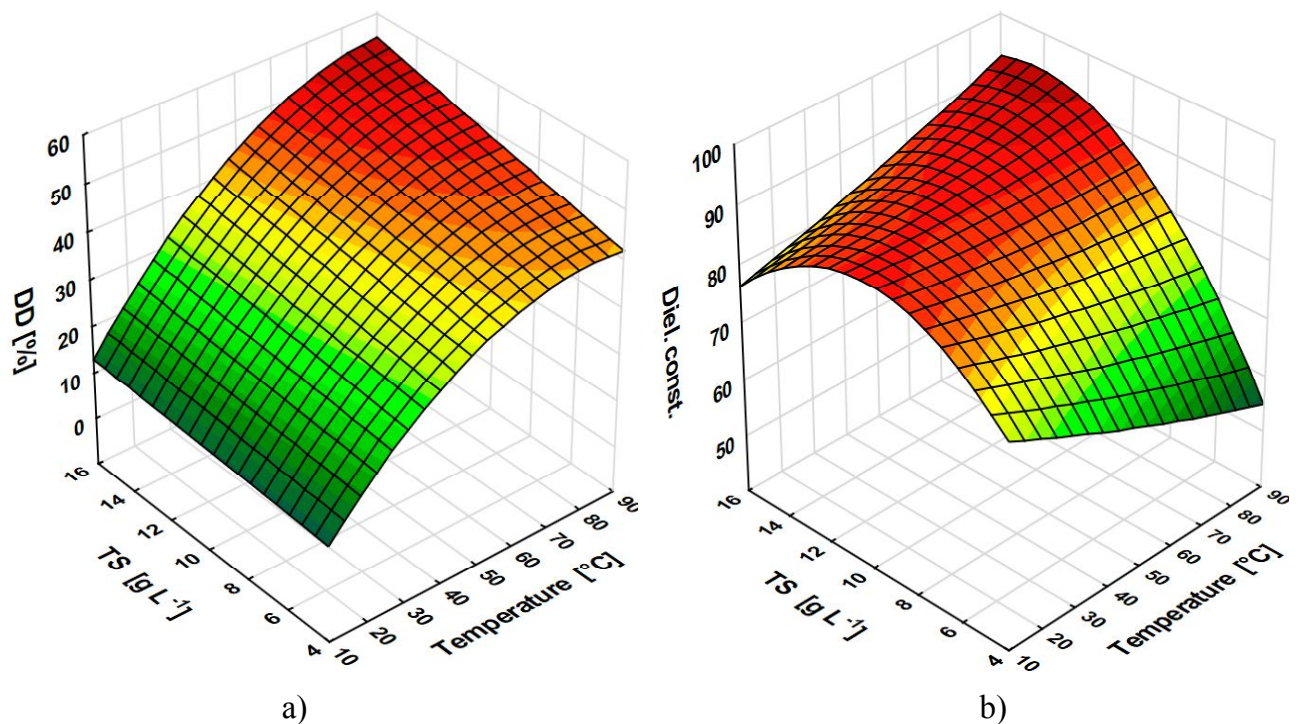


Figure 1. Disintegration degree (a) and dielectric constant as a function of TS and temperature

Figure 1 indicates that disintegration degree was mainly influenced by the pre-treatment temperature. Generally, the increment of temperature cause enhancement of DD, but over a certain value the higher temperature is not resulted in further increasing of DD (Figure1a). Due to the thermal effects the high molecular weight macromolecular components are hydrolyzed, and sludge floc and cell walls start to decompose, what increase the organic matter content in liquid phase indicated by the higher DD (Yang et al., 2013). Structural change, and hydrolysis of macromolecules led to increment of concentration of lower weight and polar components, due to the higher mobility and better polarization ability dielectric constant of pre-treated wastewater and sludge tend to increase, as well. Therefore the disintegration mechanisms are well detectable by dielectric measurement.

Considering the effects of variable TS content, different behaviour of dielectric constant can be found as a function of temperature. With temperature increasing the dielectric constant show decreasing tendency for lower TS contented samples, but increasing tendency was detected for higher solid contented wastewater sludge (Figure1b). Possible explanation for adverse effect of temperature on dielectric constant is the different composition of samples, which determine the dielectric behaviour. In lower TS contented wastewater the ratio of free water to solids is higher; therefore the dielectric behaviour is determined mainly by the relaxation mechanisms of water molecules. At the given frequency (2450 MHz) dielectric constant of water decreased with increasing temperature. If the TS content increase, the dielectric behaviour start to be more influenced by the dipolar relaxation shift of other molecules and ionic conduction of ionic compounds. Because of thermal effects, the hydrolysis of macromolecules and the release of intracellular substances have been occurred (Bohdziewicz et al., 2011). These mechanisms led to increased ratio of free water content to bound water, enhance the mobility and polarization ability of compounds in electromagnetic field led to increased dielectric constant (Brodie et al., 2014).

In order to investigate the relationship between the change of disintegration degree and dielectric parameters continuously flow microwave pre-treatments were carried out for high organic matter contented primary sludge originated from meat processing plant.

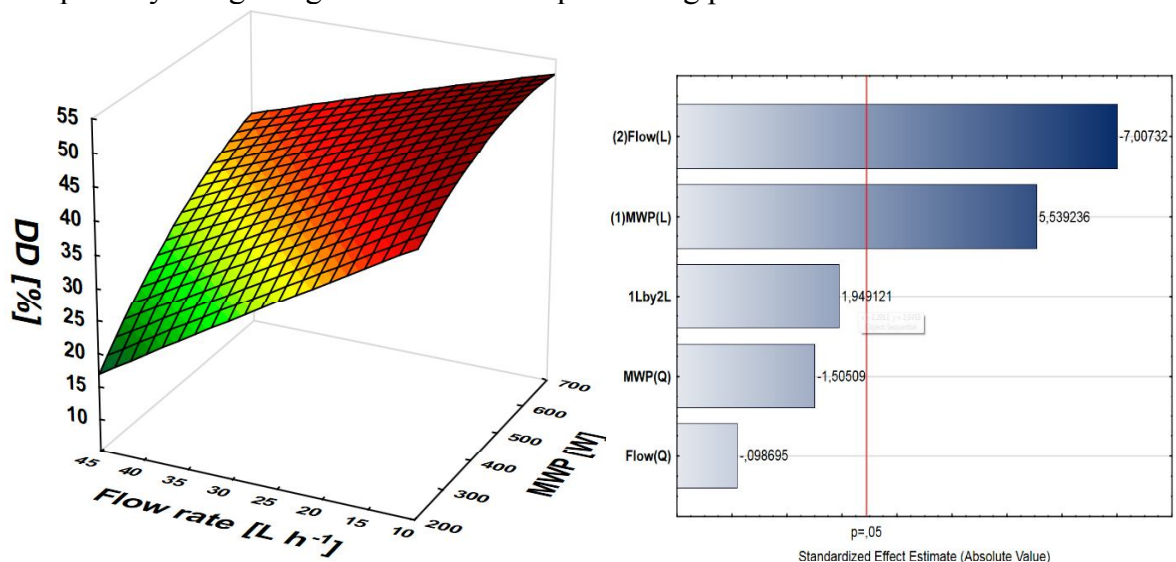


Figure 2. Change of disintegration degree of meat processing sludge during MW pre-treatments

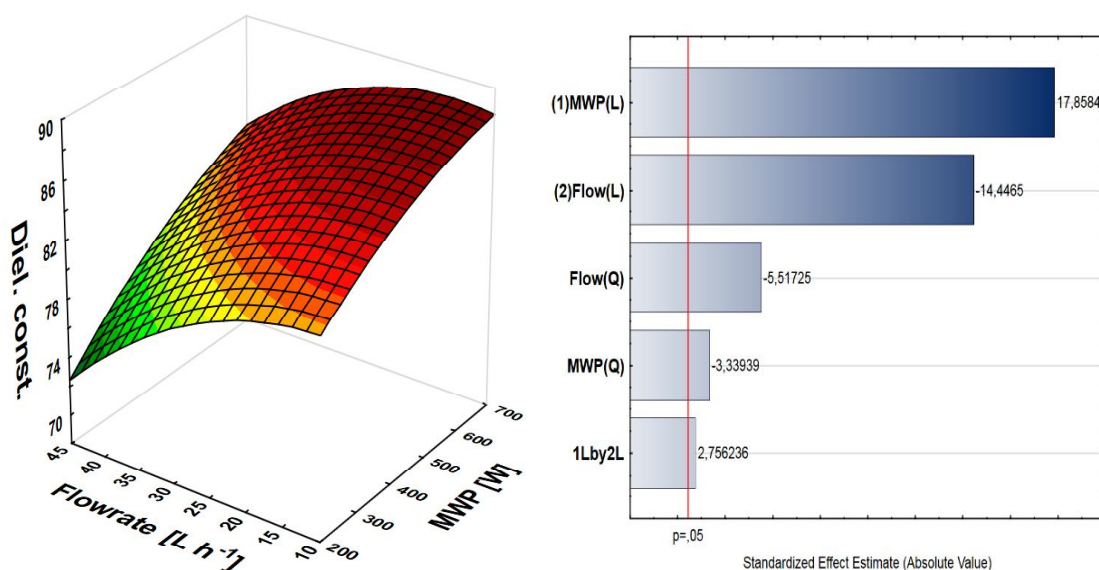


Figure 3. Dielectric constant of MW pre-treated meat processing wastewater (dielectric constant measured after cooling to temperature of 25°C for all samples)

As results show, applying continuously flow microwave pre-treatment the decreasing of volumetric flow rate (what means longer residence time in microwave reactor) and increasing of microwave power led to increased disintegration degree. Pareto chart indicates that the flow rate and microwave power has also significant effect on DD, but interaction of two variables was not significant at 95% level (Figure 3).

Dielectric constant show similar main tendency that was obtained for the DD, varying of dielectric constant indicates the physicochemical change of pre-treated sludge. Results of response surface modelling and ANOVA show that microwave power, flow rate and their interaction has also significant effect on dielectric constant of meat processing effluents, and behaviour of dielectric constant can be described by quadratic regression model. Beside that the examination of dielectric parameters are suitable to detect and quantify the structural change, the change of dielectric constant caused by microwave pre-treatment has also effect on the energetic efficiency of microwave heating.

In order to investigate the suitability of dielectric measurement for detection of changes in wastewater quality parameters, the method was tested using municipal wastewater, as well. Municipal wastewater samples came from the different stage of purification technology of a local WWTP. Figure 4 show the temperature and flow rate dependency of dielectric constant for raw wastewater (a) and for purified wastewater (b). In these experiments the dielectric constant measurements were carried out continuously flow (non static) mode to investigate the effect of flow rate on dielectric parameters and to test the sensitivity of measurement method for sample homogeneity.

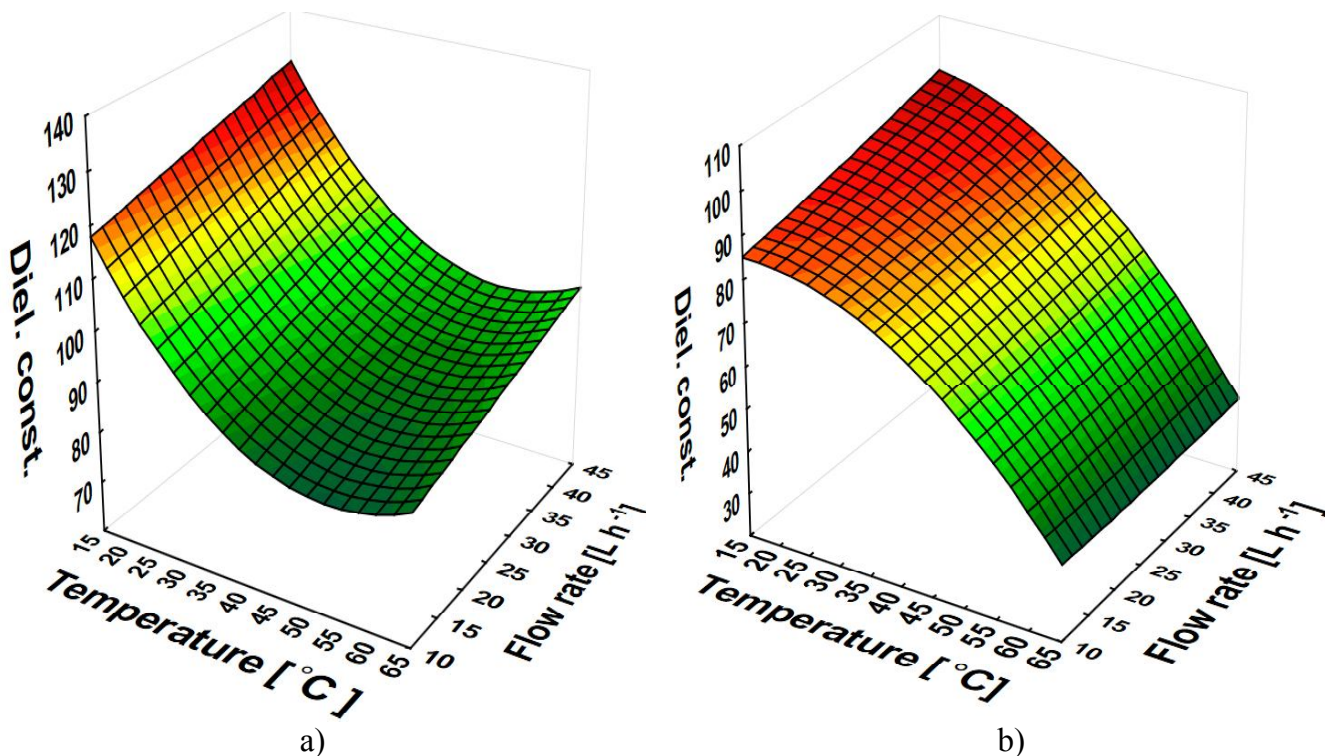


Figure 4. Dielectric constant of raw (a) and purified (b) municipal wastewater

Results indicated that the change of dielectric constant versus volumetric flow rate and temperature was different for raw and purified municipal wastewater (Figure 4). Because of high particulate matter content of raw wastewater, especially at higher temperature range the flow rate has effect on the dielectric constant. Due to the higher temperature, structural changes has been occurred in raw wastewater, and higher velocity assist disruption of cell walls, and because of higher homogeneity enhance the efficiency of hydrolysis of macromolecular components and disintegration of particulates. These effects led to higher value of dielectric constant. Considering the effect of temperature, it can be found a break point (above range of 50-55°). The increment of dielectric constant above a critical temperature range was due to thermal hydrolysis of macromolecular components and release of intracellular substances into the intercellular space. These effects can be more expressed if organic matter content of wastewater is higher (see Figure 1b).

In the case of lower organic matter contented purified water, the effect of flow rate on dielectric constant at a given temperature was slight and non significant, and the temperature dependency had similar tendency that of known for 'pure' water (Holtze et al., 2006). It can be summarized, that the measurement of dielectric constant is suitable method to detect the change in organic matter removal efficiency during a conventional wastewater purification technology.

CONCLUSION

Results of this research on the dielectric constant measurement for investigation of wastewater treatment process and disintegration of particulate organic matters support the finding that dielectric measurements are suitable to detect and quantify the physicochemical changes and organic matter removal during a purification process. Although the main component of wastewater is the water, the dielectric behaviour as a function of temperature or the volumetric flow rate was different that of obtained for pure water. Thermal pre-treatments have effect on disintegration of particulates in municipal and food industry wastewater, and these changes have effect on dielectric constant, as well. Our preliminary results make suitable to develop real time and in-line dielectric measurement methods to detect the disintegration and removal efficiency of wastewater purification process.

Furthermore the change of energetic efficiency during microwave treatments can be controllable by the application of dielectric measurements.

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